Design-of-Experiment Approach to Hydrogen Re-Embrittlement Evaluation

WP - 2152

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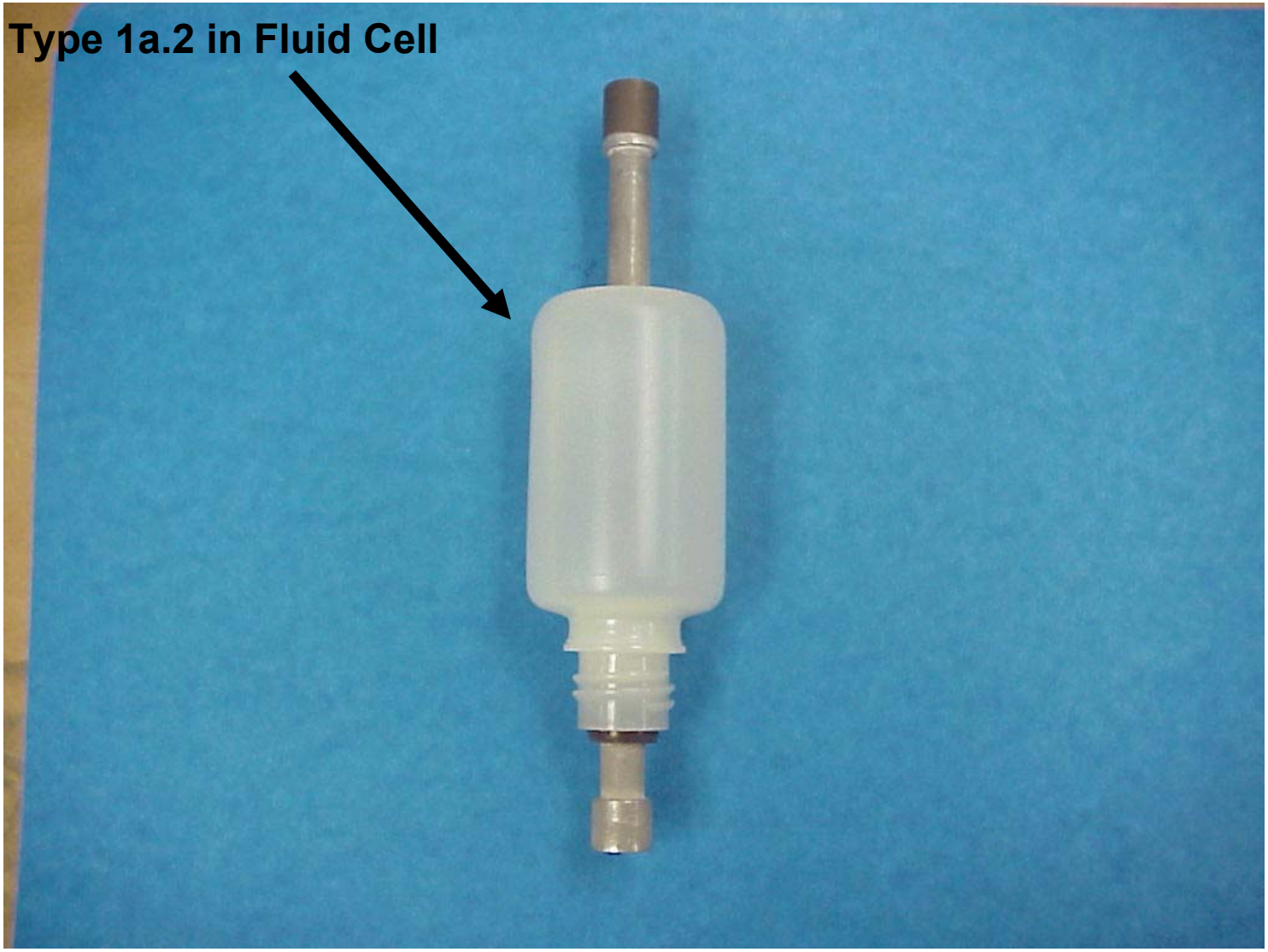
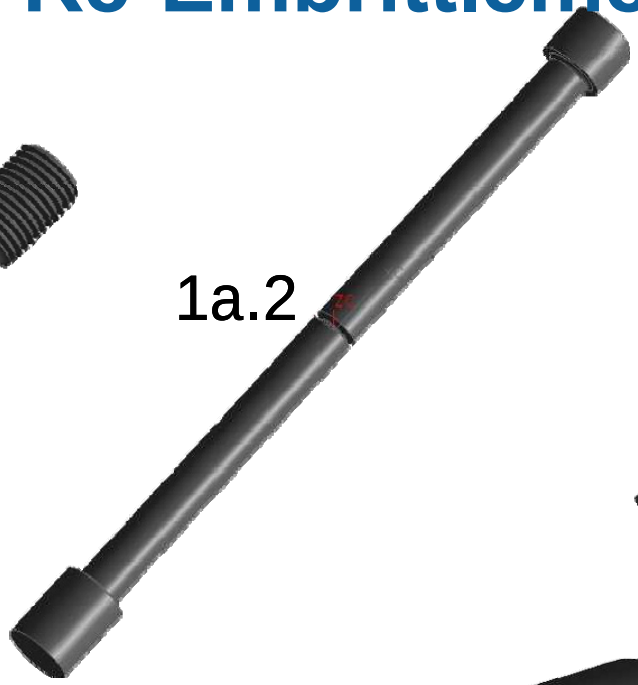
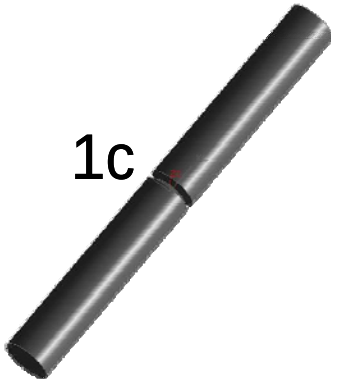
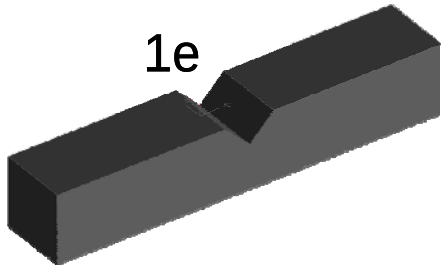

Type 1a.2 in Fluid Cell

Re-Embrittlement Test Issues







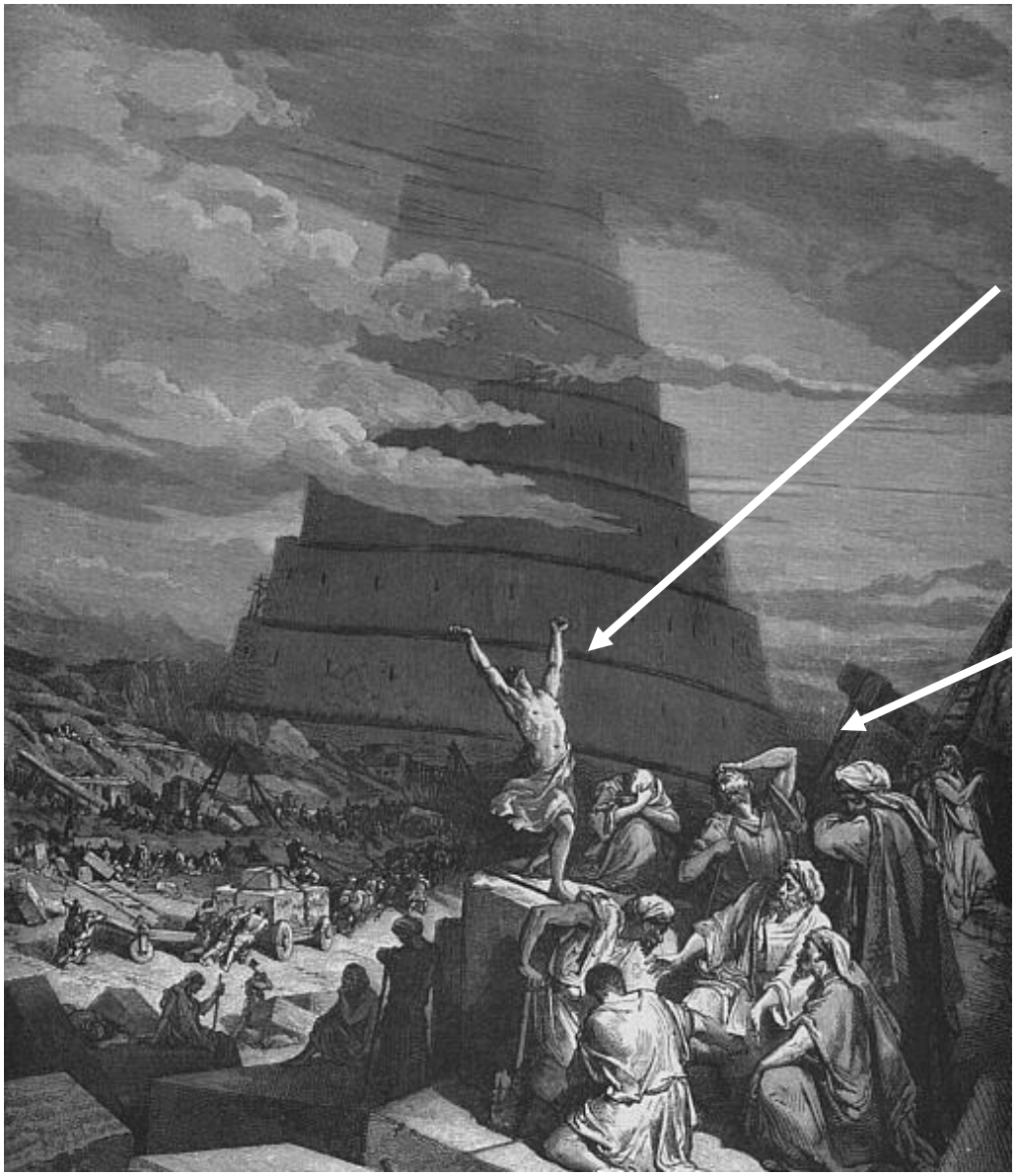
Re-Embrittlement Test Issues

- Not Standardized Across the Industry – **Variations of ASTM Annex A5 Used**
  - Various ASTM F519 Specimens Used
    - Type 1.a.1, 1.a.2, 1b, 1c, 1d, 1e, and 2a
  - Various Specimen Immersion Methods
    - Wet for 150 hrs, Wet Then Dry, Concentrated or Diluted Chemicals
    - Volume of Fluid, Temperature
  - Various Loading Methods
    - Tension, Bending, Sustained Load, Incremental Step Loading (24 hours)
    - 45%,65%,75% NFS, 80%YS for 150 or 200 hrs
  - Various Strength Levels to Bake or No-Bake
    - 160 ksi, 180 ksi, 200 ksi, 220 ksi

Re-Embrittlement Testing Is Our Tower of Babel

The Confusion of Tongues by Gustave Doré (1865)

Typical ASTM F07.04 Hydrogen Embrittlement Meeting



Project Team

- Lead PI – S. Grendahl, ARL
- E. Babcock, S. Gaydos, J. Osborne, S. Zhu – The Boeing Co.
- C. Willan – Omega Research Inc.
- J. Green – NAVAIR Pax River
- C. Hogan – Hill AFB
- R. Green – Green Specialty Service
- D. Kelly – ASKO Processing Inc.

Technical Objective

- **Increase the implementation and utilization of environmentally friendly maintenance chemicals and cadmium alternatives by alleviating the HE obstacle.**
- **Year 1 – Life models for aerospace grade 4340 steel**
- **Year 2 – Life models for prospective maintenance chemicals**
- **Year 3 – Life models for prospective alternative coatings**

Technical Approach

- He testing has traditionally been done pass/fail on worst case material

- DoE approach develops life prediction models over a range of material strength, applied stress, and environment
  - ♦ 280 ksi
  - ♦ Stress varies with geometry
  - ♦ Cad plated steel

Vs.

- 140 - 280 ksi
- 10 - 95% NFS
- % of NaCl, or Conc. or Plating

- Statistical analysis allows a reasonable matrix size while accounting for full spectrum of variables with prediction.
  - ♦ 5x5x5x5 (625) Vs. ● 400

Technical Approach

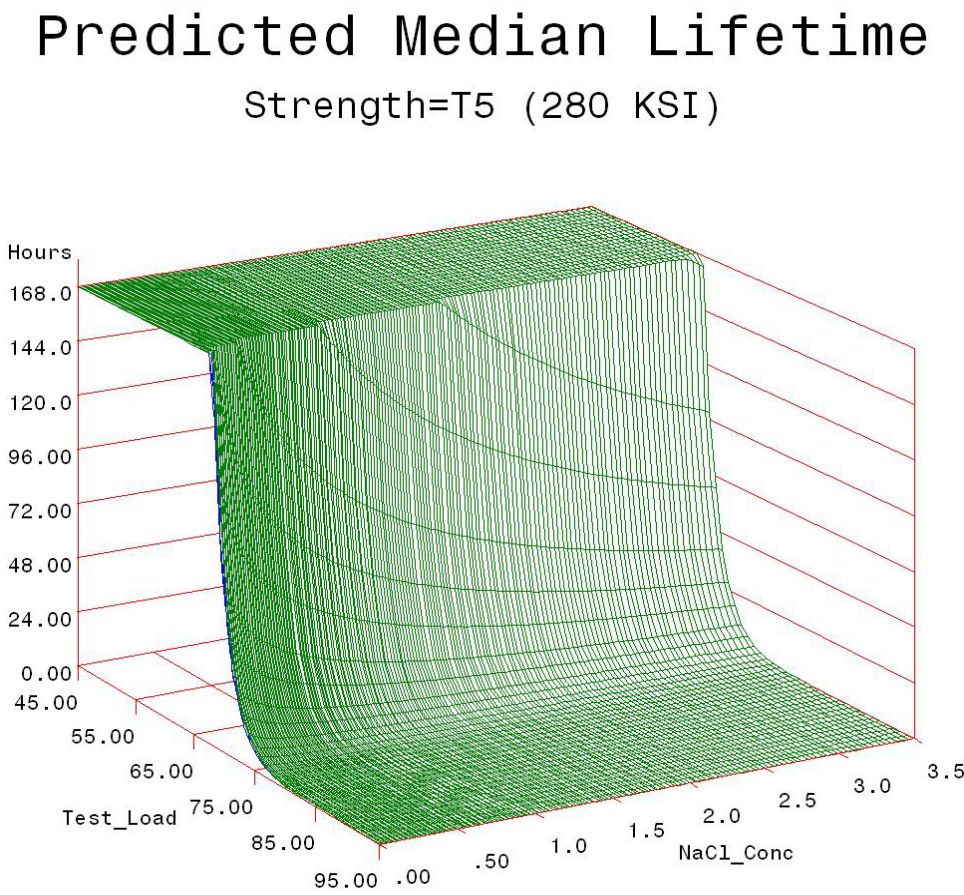
Condition	-α	-	0	+	+α
Strength (ksi)	140	158	210	262	280
Test Load (% NFS)	40	45	60	75	80
NaCl Concentration (wt% NaCl)	1.25E-05	0.01	0.50	2.36	3.5

- Linear with Center points
- Quadratic
- Confirmation Runs

- Base model is developed from Linear and Quadratic portions
  - Y=ln X =19.01 –11.67*strength -9.93*test_load -0.88*NaCl + error
  - Run confirmations, re-compute, and refine model

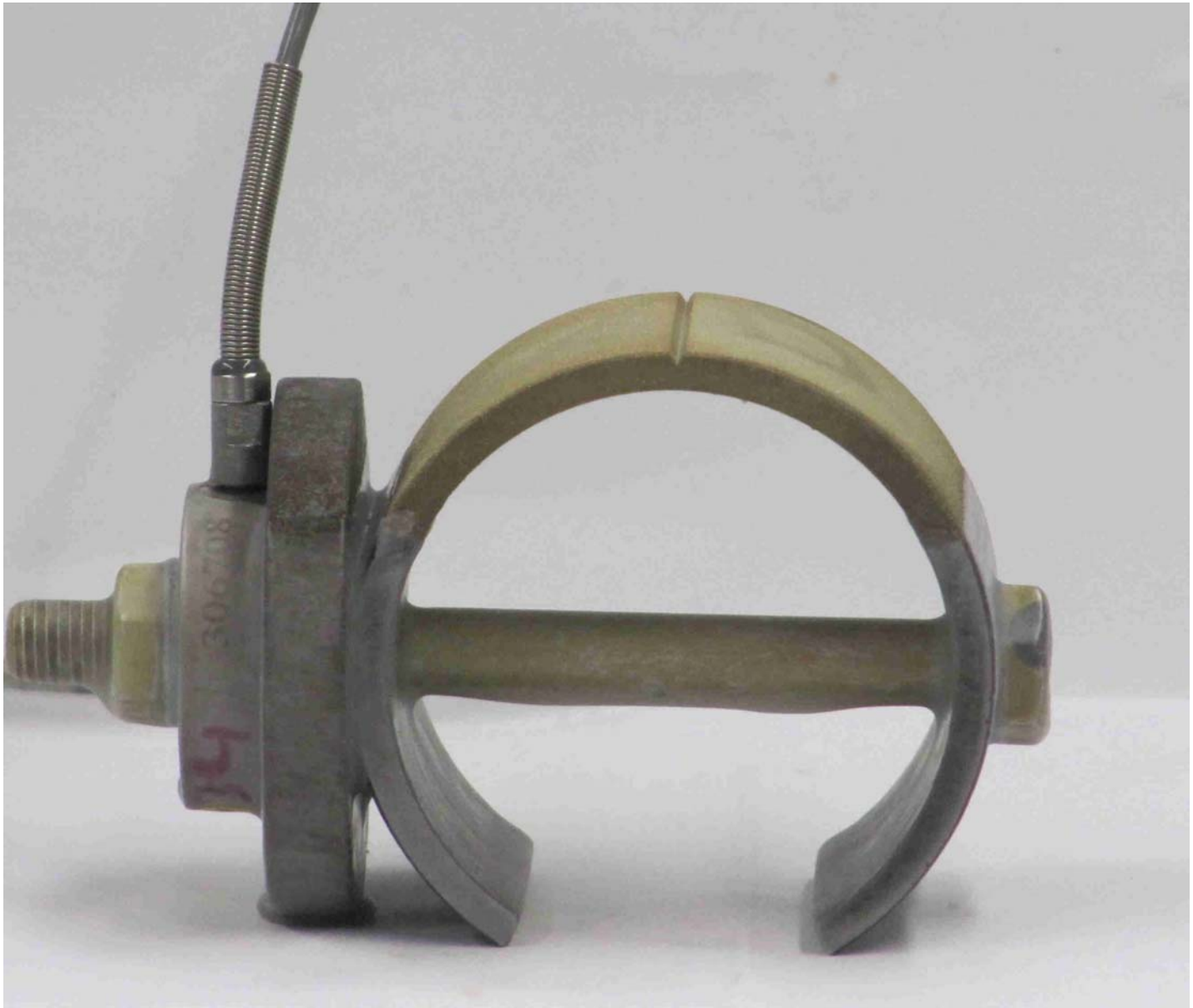
Technical Approach

- Material Strength (140 -280 ksi)
- Applied Stress (% of NFS)
- Environment (wt% of NaCl, or Conc. of chemical or thickness of coating
- Model Yields - TTF (Time to Failure)

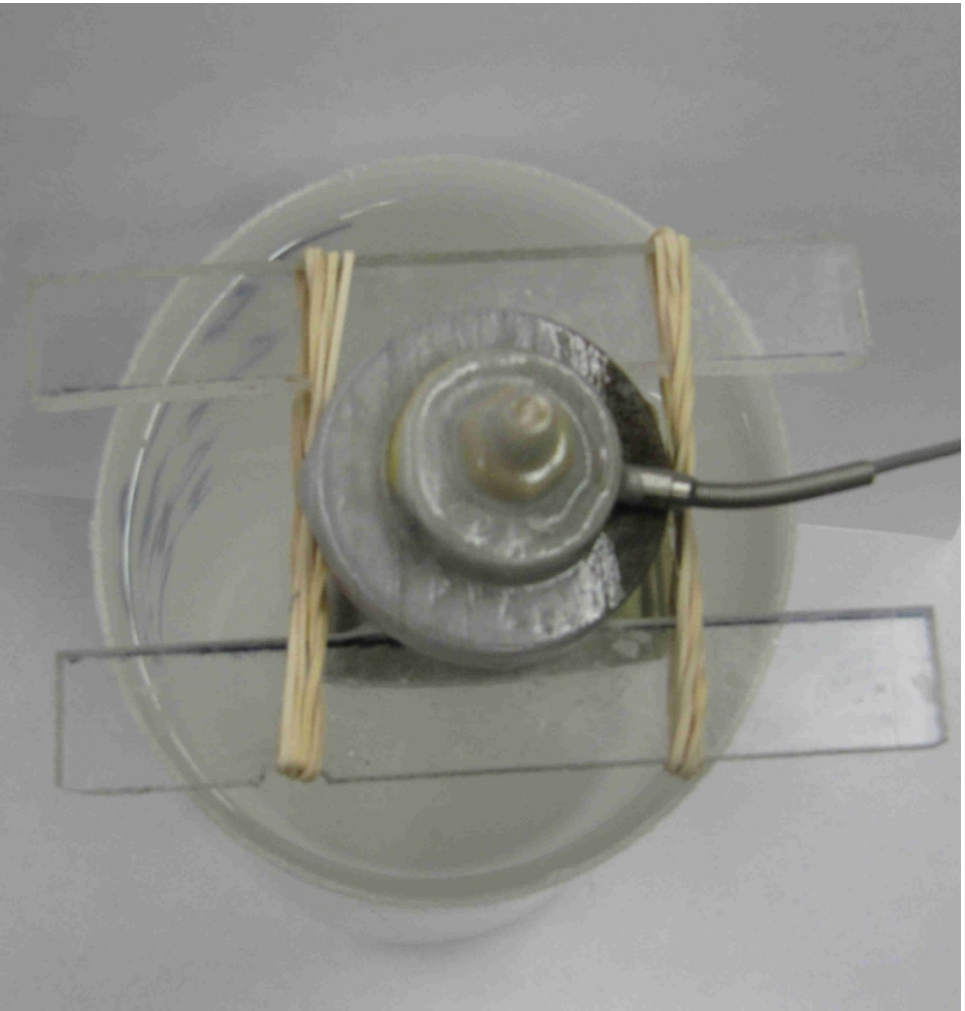


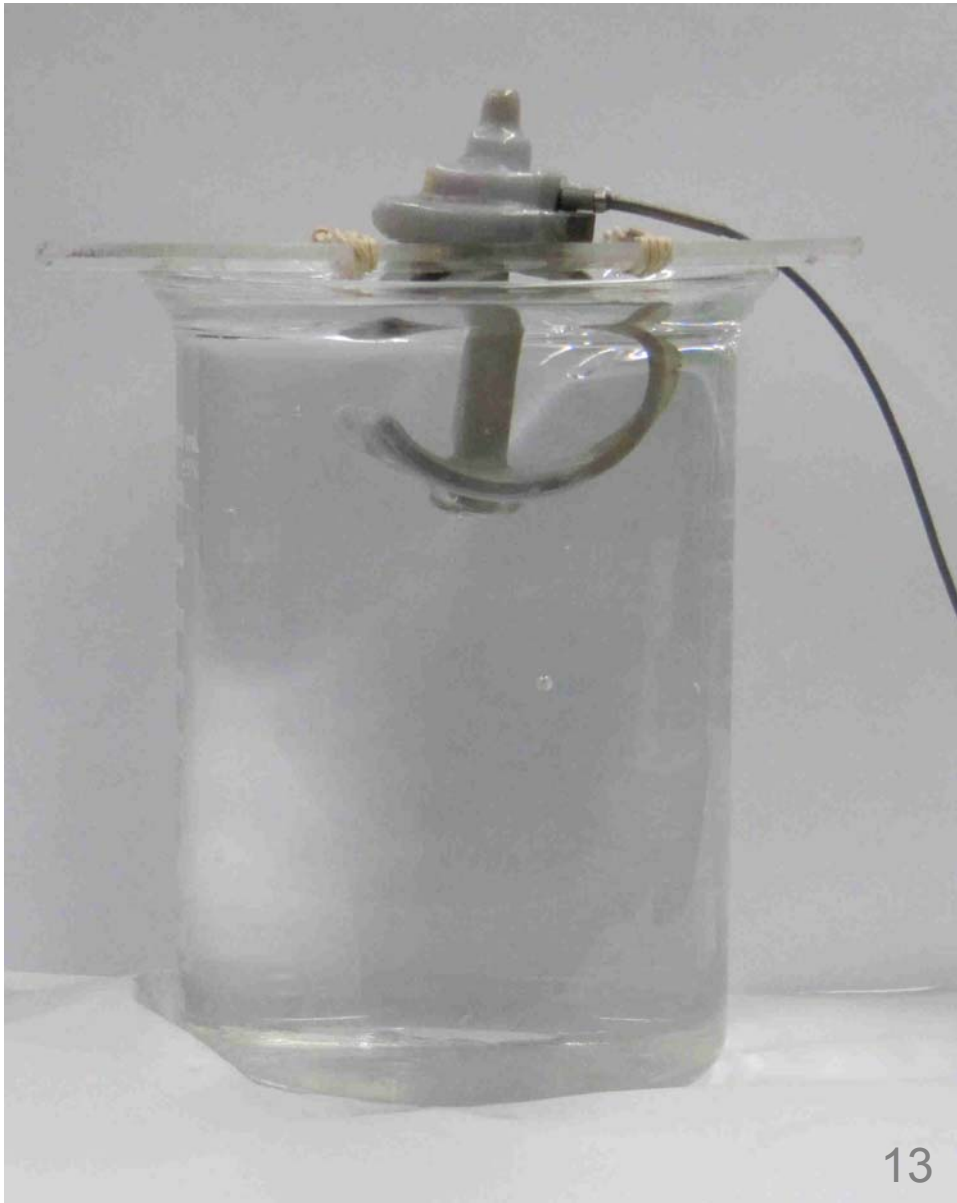
ASTM F 519 Type 1c Original

Technical Approach



Technical Approach





Technical Approach

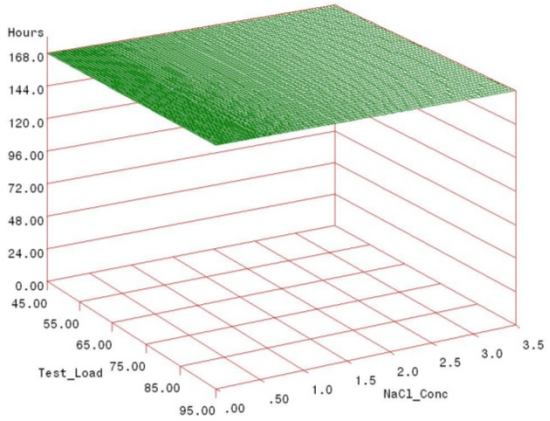
- Year 1 models will be created for aerospace 4340 steel
- Assess results via team to determine best geometry for program
- 1 geometry then used to assess applicable maintenance chemicals or platings
- Results will provide the airworthiness authority data to assess which processes (chemicals or platings) and applications are safe zones.

Seed Funding Results

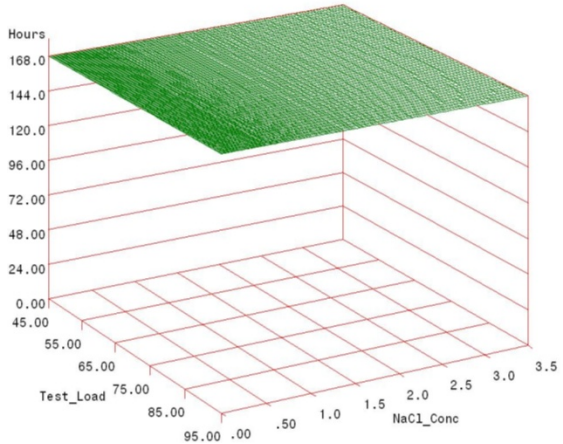
- **Air-melted 4340 steel, 10 - 95% NFS, 0 - 3.5% NaCl**
- **5 geometries, 5 heat treatments**
- **Separate models for each heat treatment (or material strength)**
  - *T1 140 ksi*
  - *T2 158 ksi*
  - *T3 210 ksi*
  - *T4 262 ksi*
  - *T5 280 ksi*

1a1 Results

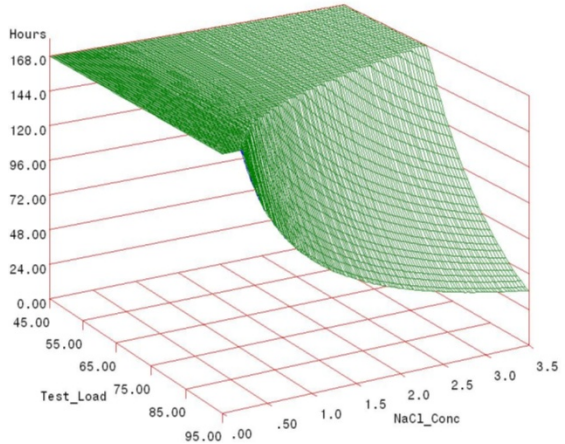
Predicted Median Lifetime
Strength=T1 (140 KSI)



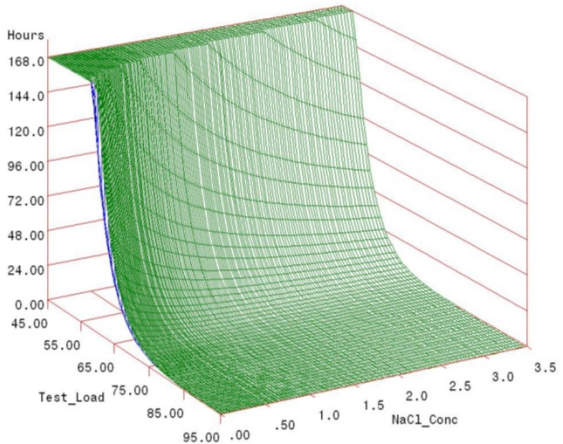
Predicted Median Lifetime
Strength=T2 (158 KSI)



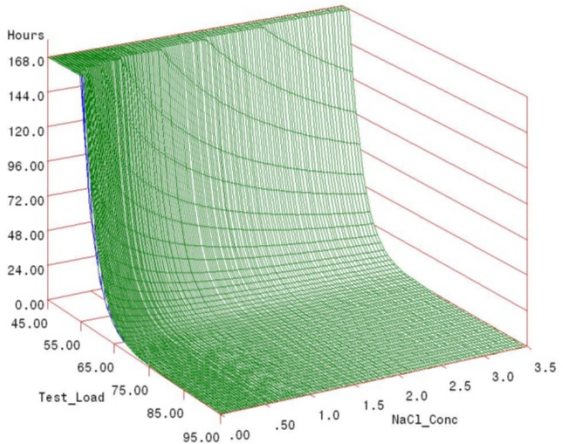
Predicted Median Lifetime
Strength=T3 (210 KSI)



Predicted Median Lifetime
Strength=T4 (262 KSI)



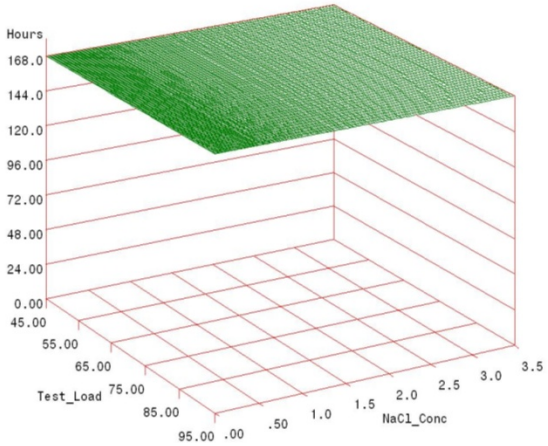
Predicted Median Lifetime
Strength=T5 (280 KSI)



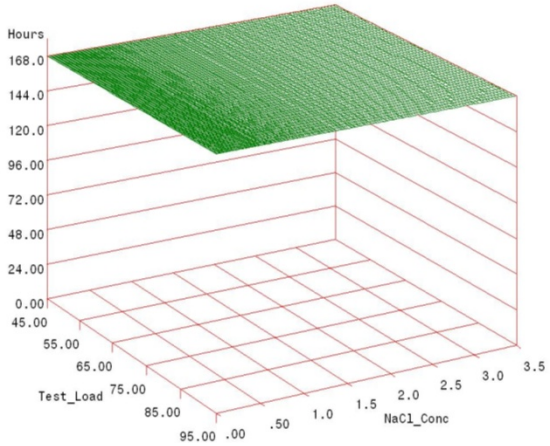
T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi

1a2 Results

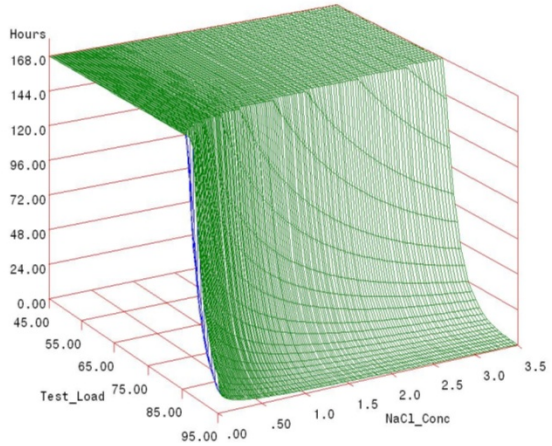
Predicted Median Lifetime
Strength=T1 (140 KSI)



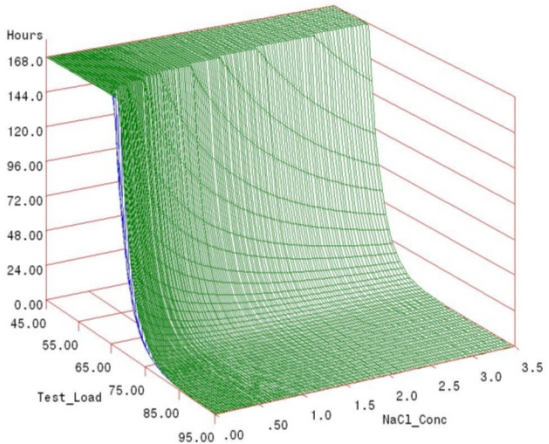
Predicted Median Lifetime
Strength=T2 (158 KSI)



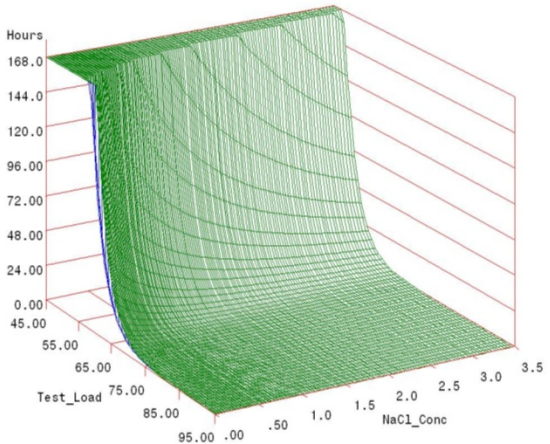
Predicted Median Lifetime
Strength=T3 (210 KSI)



Predicted Median Lifetime
Strength=T4 (262 KSI)



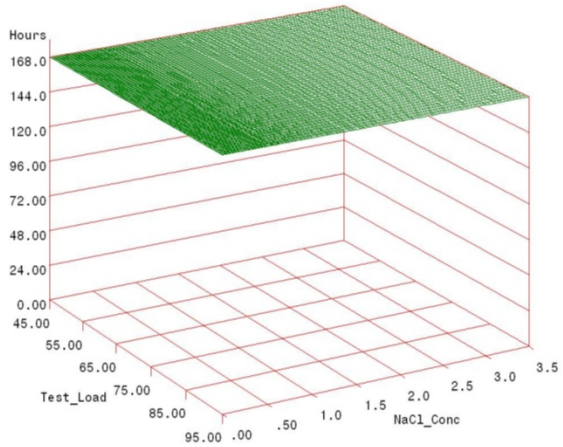
Predicted Median Lifetime
Strength=T5 (280 KSI)



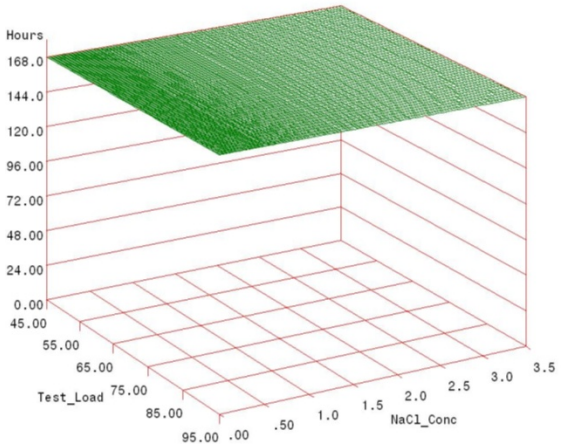
T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi

1c Results

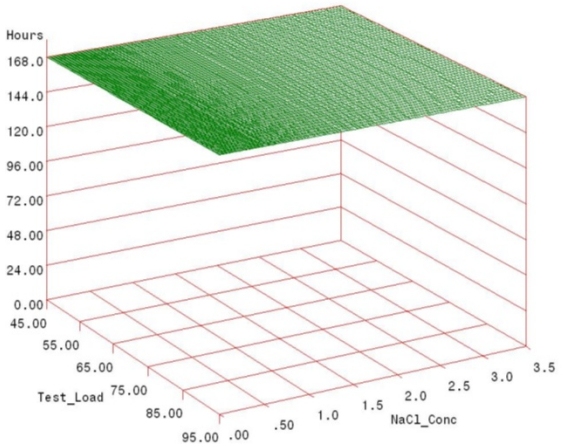
Predicted Median Lifetime
Strength=T1 (140 KSI)



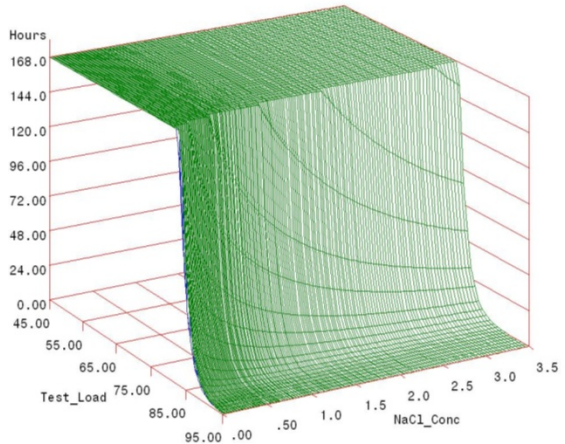
Predicted Median Lifetime
Strength=T2 (158 KSI)



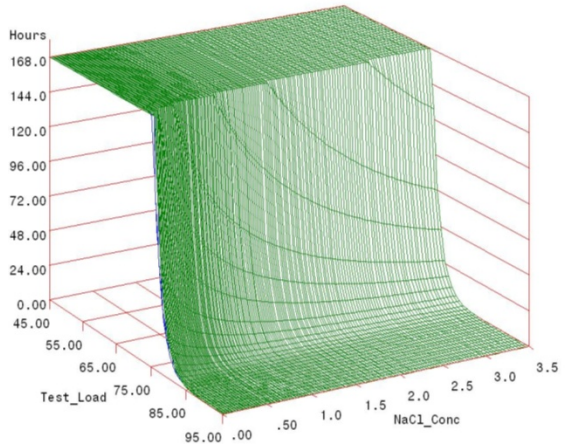
Predicted Median Lifetime
Strength=T3 (210 KSI)



Predicted Median Lifetime
Strength=T4 (262 KSI)



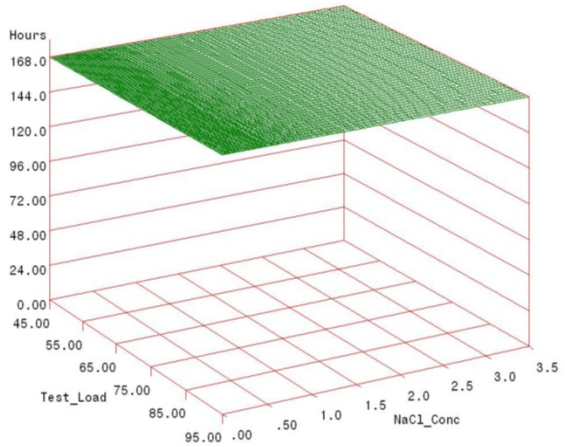
Predicted Median Lifetime
Strength=T5 (280 KSI)



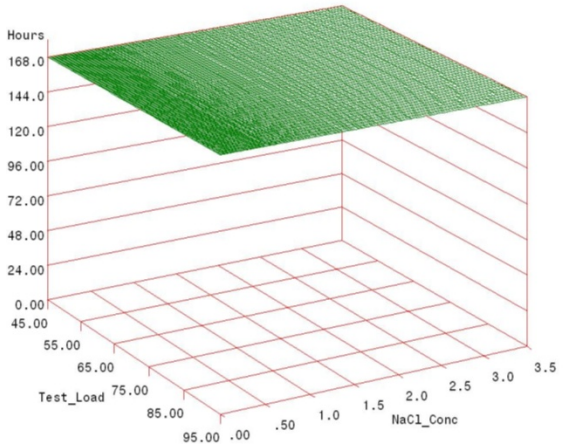
T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi

1d Results

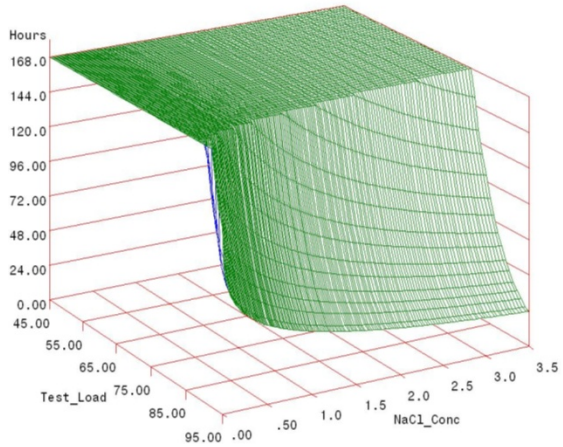
Predicted Median Lifetime
Strength=T1 (140 KSI)



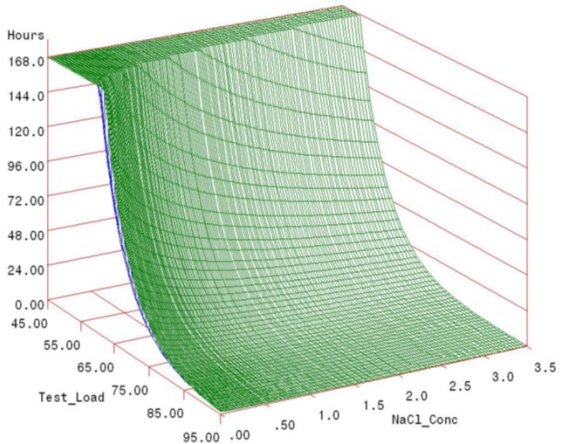
Predicted Median Lifetime
Strength=T2 (158 KSI)



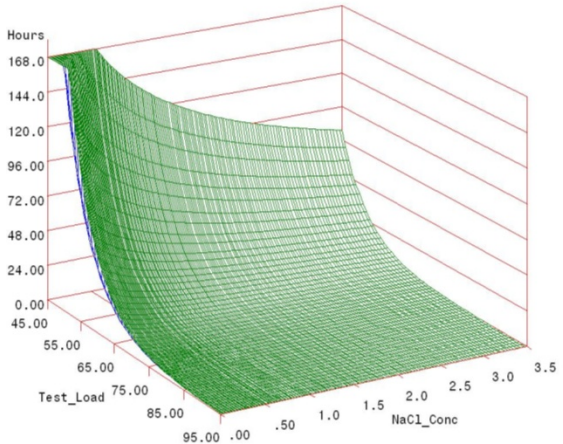
Predicted Median Lifetime
Strength=T3 (210 KSI)



Predicted Median Lifetime
Strength=T4 (262 KSI)



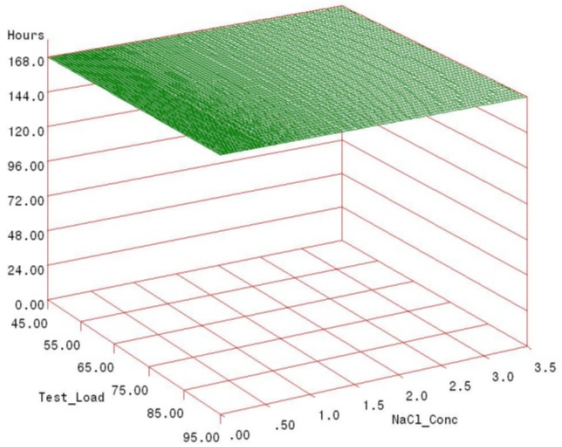
Predicted Median Lifetime
Strength=T5 (280 KSI)



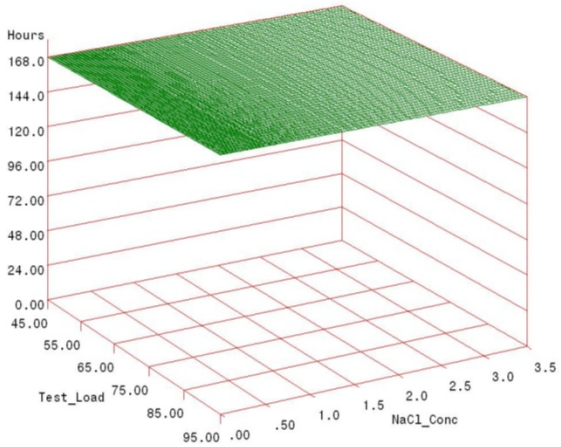
T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi

1e Results

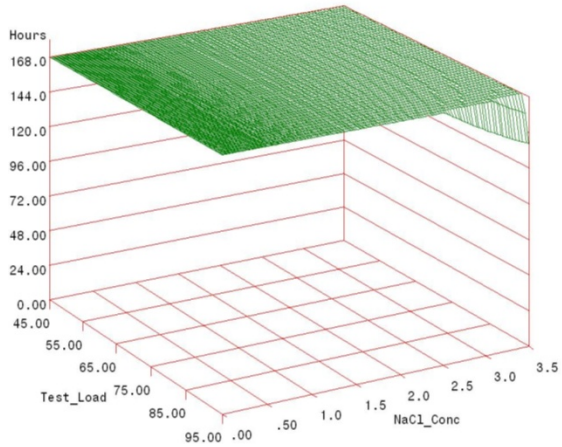
Predicted Median Lifetime
Strength=T1 (140 KSI)



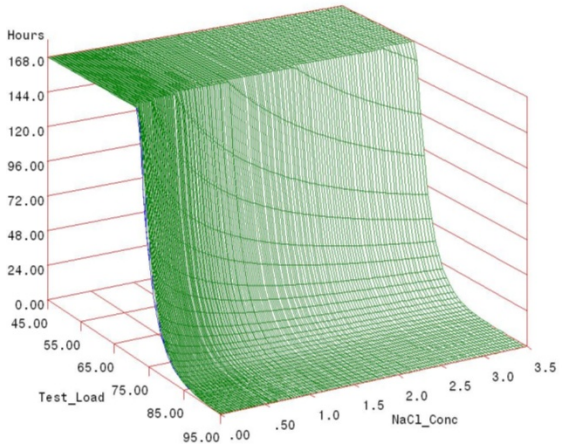
Predicted Median Lifetime
Strength=T2 (158 KSI)



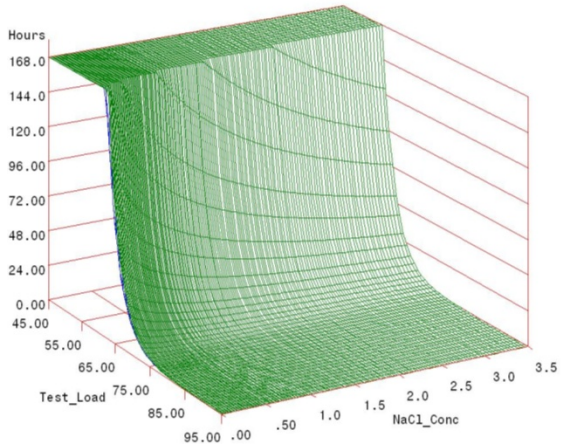
Predicted Median Lifetime
Strength=T3 (210 KSI)



Predicted Median Lifetime
Strength=T4 (262 KSI)



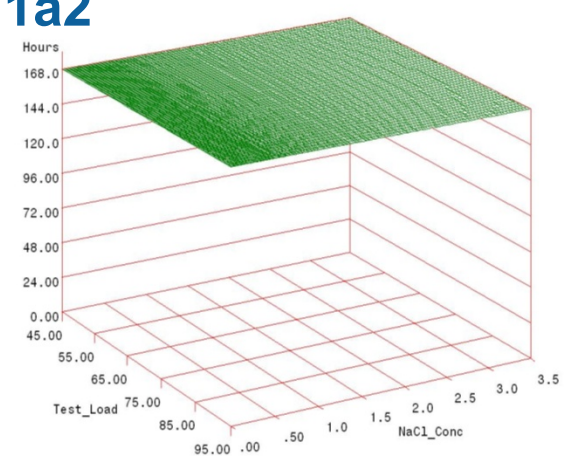
Predicted Median Lifetime
Strength=T5 (280 KSI)



T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi

T2 (158 ksi) Results

1a2



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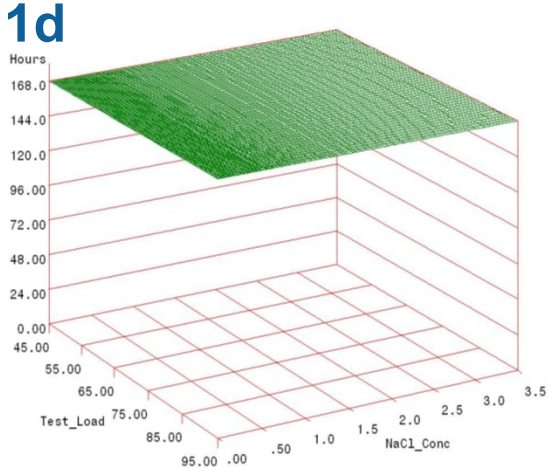
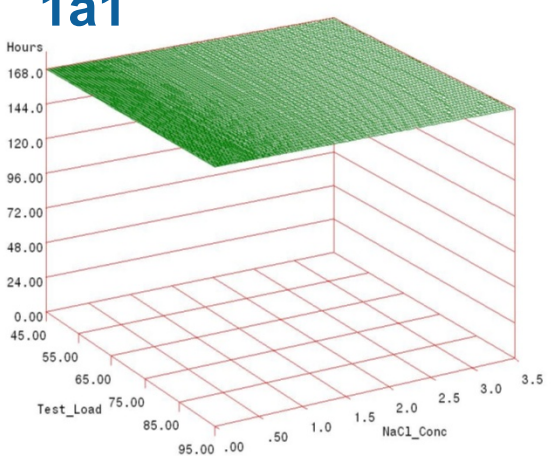


1a1



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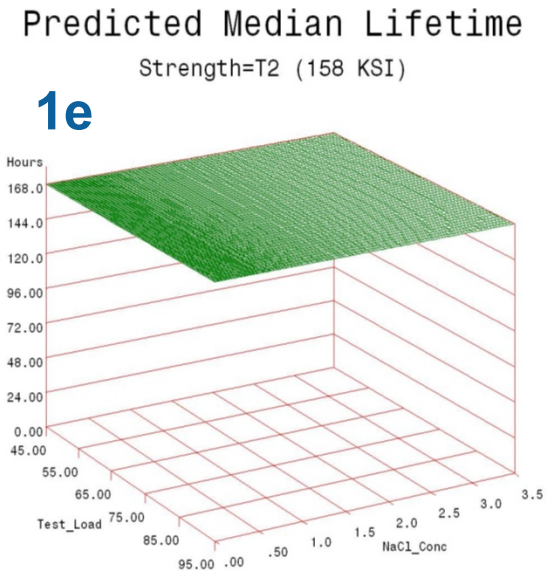

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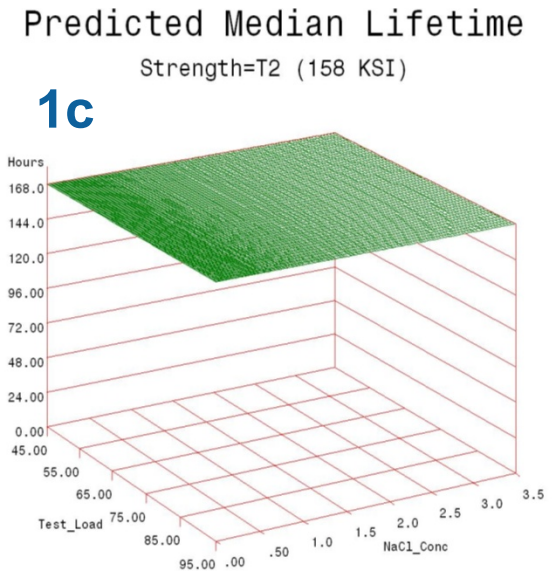
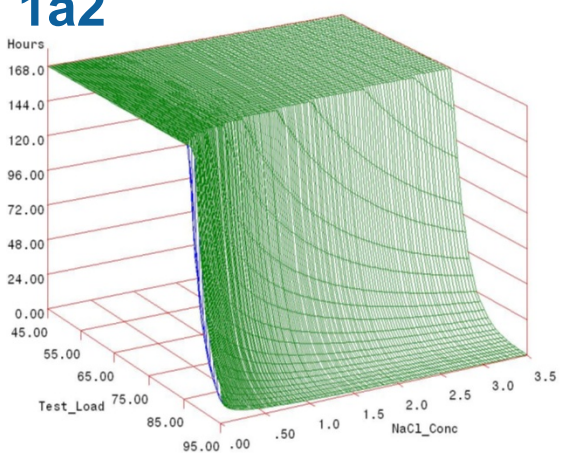


T3 (210 ksi) Results

1a2



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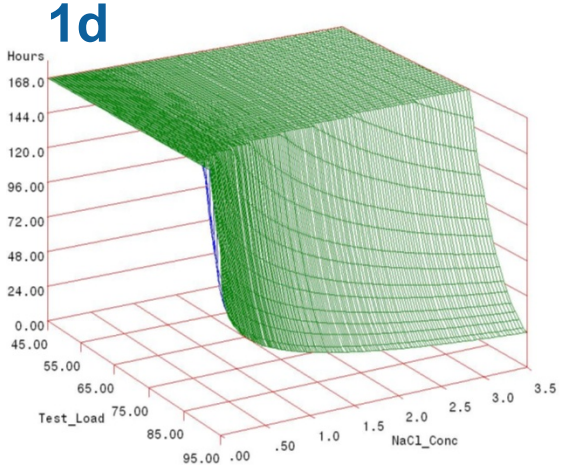
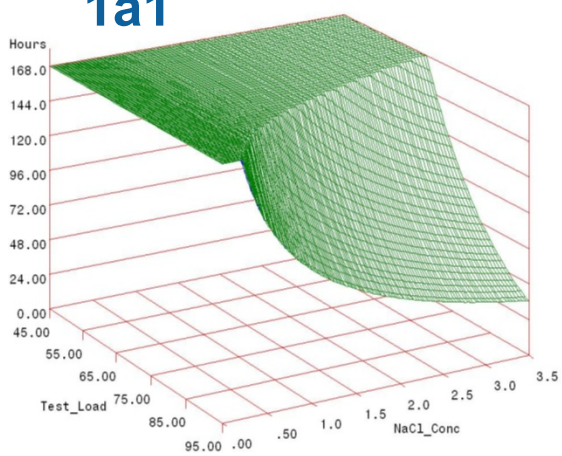


1a1



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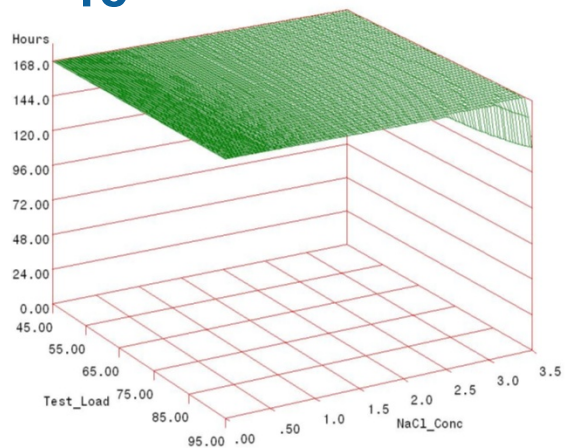

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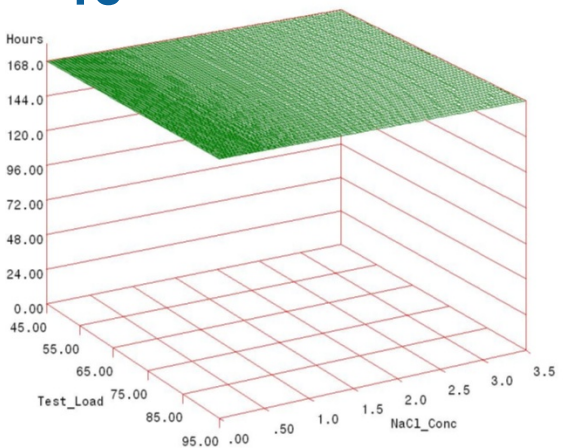
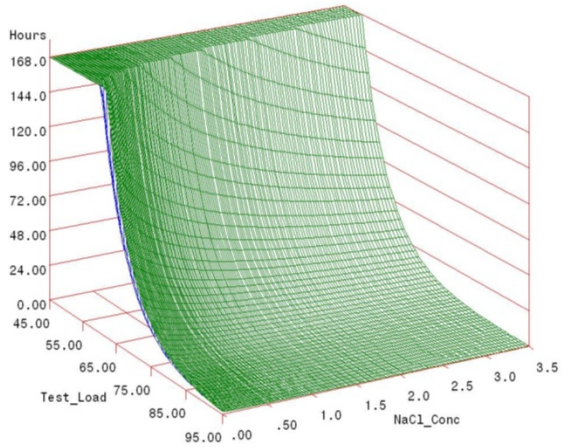


T4 (262 ksi) Results

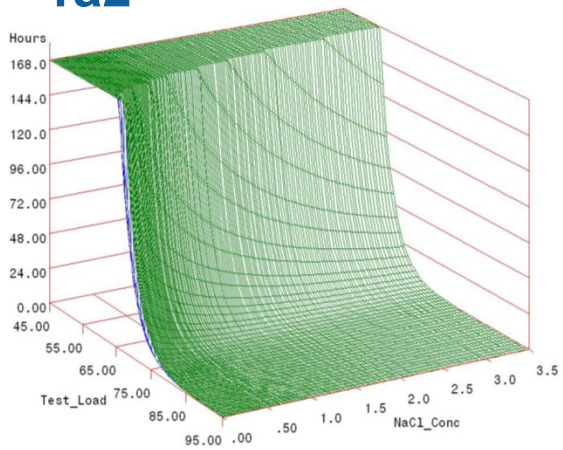
Predicted Median Lifetime
Strength=T4 (262 KSI)

1d



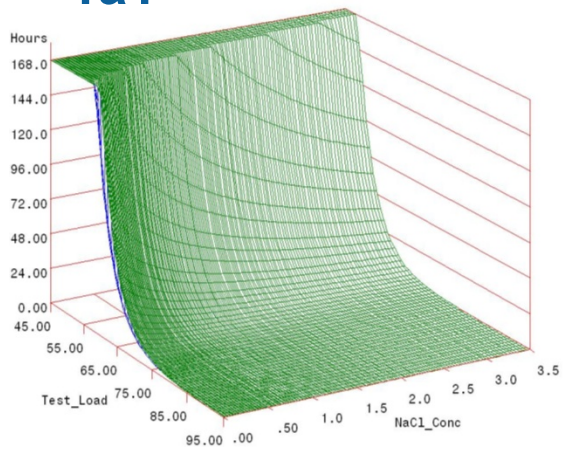
Predicted Median Lifetime
Strength=T4 (262 KSI)

1a2



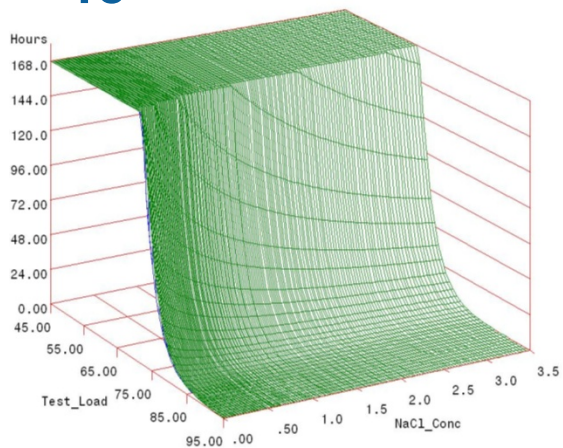
Predicted Median Lifetime
Strength=T4 (262 KSI)

1a1



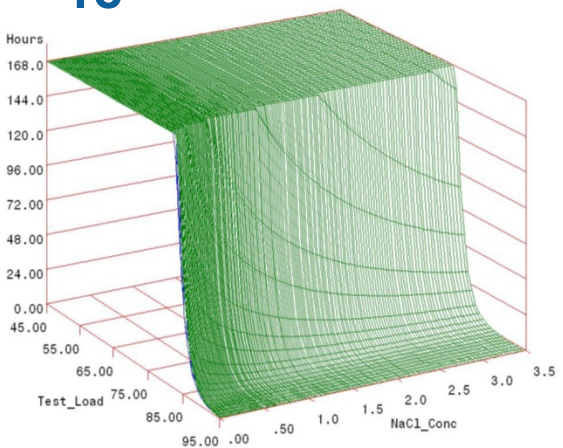
Predicted Median Lifetime
Strength=T4 (262 KSI)

1e



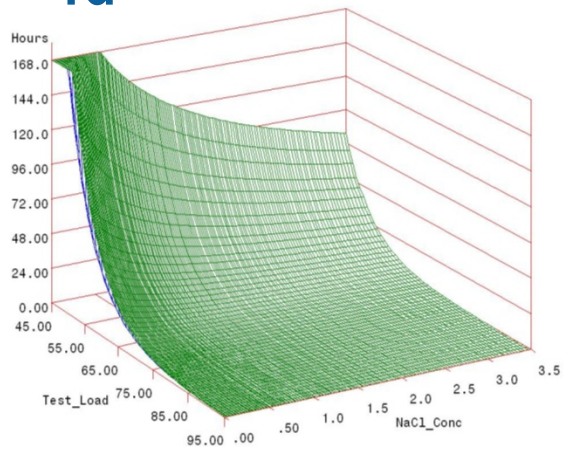
Predicted Median Lifetime
Strength=T4 (262 KSI)

1c

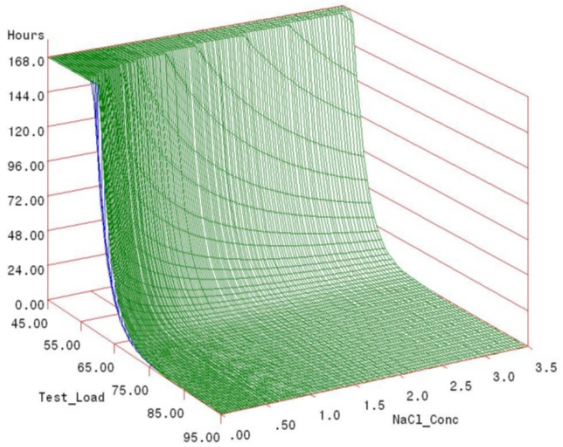


T5 (280 ksi) Results

1d



1a2



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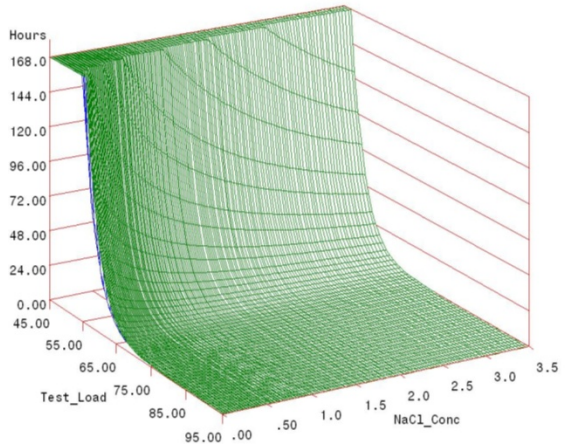
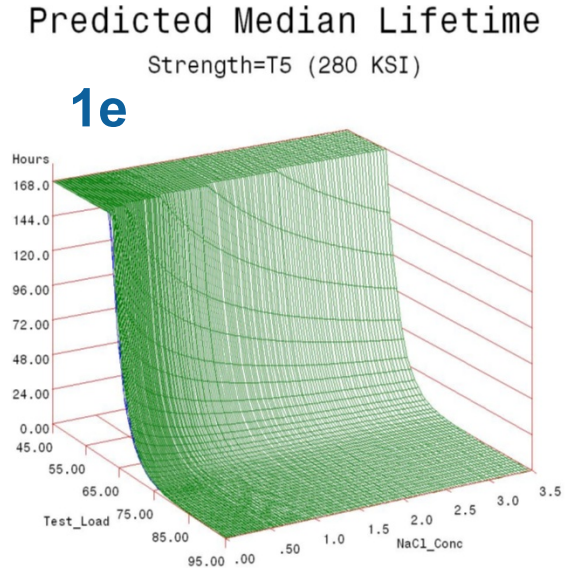
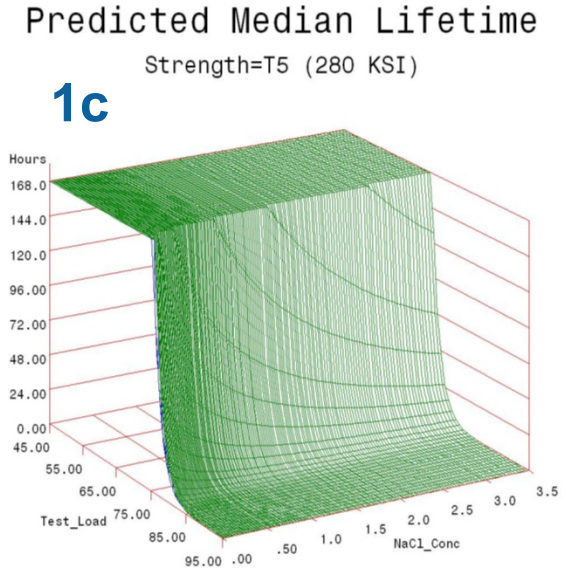


1e



1c



Transition Plan

- Work has been briefed and discussed by ASTM committee F07 on Aerospace and Aircraft, and in detail within subcommittee F07.04 on hydrogen embrittlement
- Most active participants of the committee are directly involved
- Changes to F-519 are likely upon completion and data review
- Lifetime prediction models for the targeted maintenance chemicals will be utilized by AMCOM/AMRDEC to alleviate the presently existing requirement of bake relief treatments for processes that have failed HE testing
  - Material applications below susceptibility threshold (e.g. 180 ksi)
  - Service stress applications below threshold (e.g. below 50% UTS)

- Lifetime prediction models for cadmium alternatives will be transitioned to service use for applications shown to be below the HE susceptibility threshold (e.g. ZnNi below 1.5 mils on 200 ksi steel)
- Commercial partners will follow guidance from the aviation authority in implementing targeted applications deemed safe.

BACKUP MATERIAL

Acronyms and Symbols

- **HE - Hydrogen Embrittlement**
- **NFS - Notch Fracture Strength**

Prior and Leveraged Work

- Boeing Ruggedness Study
  - Aimed at establishing which factors were most important
    - Surface condition plated or bare
    - Notch condition plated or bare
    - Solution Volume
    - Solution Temperature
    - Solution Concentration
    - Exposure Time
    - Exposure Temperature
- Boeing Risk Reduction Study
  - 1a1 and 1d geometries at 519 strength and load levels
  - Assessment of NaCl solution merit, low strength material procedure
- SPOTA/ARL for re-machining and Aerospace Grade material purchase
- ASTM Committee and coordination work - unfunded
- ASKO Plating for developmental work
- Boeing and ARL labor to date

Publications

- Barron, J., “Effect of Coatings on the Structural Integrity of Fasteners”, ASTM F16.96 workshop, Northrop Grumman Shipbuilding, Newport News, 20 May 2009.
- Gaydos, S., “ASTM F 519 Annex A5 DoE Test Plan Status”, Presentation at ASTM F07.04 Subcommittee Meeting, The Boeing Company, St. Louis, MO, 15 April 2008.
- Babcock, E. A., “Aqueous Cleaning of High Strength Steel”, Whitepaper WP 3M11:07-051, To AED, The Boeing Company, Mesa, Arizona, 24 May 2007
- Gaydos, S., “SERDP Hydrogen Re-Embrittlement DoE Test Plan Status”, Presentation to DoD Metal Finishing Workshop – Chromate Alternatives for Metal Treatment and Sealing, The Boeing Company, St. Louis, 17 May 2007
- Babcock, E. A., “Update on ASTM F 519”, Presentation to AMCOM G-4 OEM at Redstone Arsenal, The Boeing Company Mesa, Arizona, April 24-25, 2007
- Babcock, E. A., “Annex 5 Ruggedness DoE Results + SERDP”, Presentation to ASTM International Committee F07.04 on Hydrogen Embrittlement, The Boeing Company, Mesa, Arizona, 17 April 2007
- Babcock, E. A., “Codifying Hydrogen Embrittlement Testing Protocols - Sound Tools for Alternatives Testing”, Presentation at Seventeenth Annual Cleaner Sustainable Industrial Materials & Process (CSIMP) Workshop, The Boeing Company, Mesa, Arizona January 21, 2007
- Babcock, E. A., “Hydrogen Embrittlement Testing and Evaluation: Progress and Status of Ongoing Research and Development”, Presentation to ASTM International Committee F07.04 on Hydrogen Embrittlement, The Boeing Company, Mesa, Arizona, 15 November 2006.